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READOUT AND CONTROL SYSTEM FOR A DIRECT READING OPTICAL EMISSION SPECTROMETER PART I: THE HARDWARE

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READOUT AND CONTROL SYSTEM FOR A DIRECT READING OPTICAL EMISSION SPECTROMETER

PART I: THE HARDWARE

by

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ABSTRACT

This report is a chronological summary of the stepwise hardware changes which were made to an optical emission spectrometer to convert it to a computer controlled system. The converted system provides a decreased analysis time, improved reproducibility and on-line data handling with a minimum of operator intervention. Part II will be a detailed description of the computer program which controls the operation of the system. Part III will be an operators manual which will provide the detailed instructions for the operation of the system to provide a sample analysis and analytical report.

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SYSTÈME D'AFFICHAGE ET DE COMMANDE D'UN SPECTROMÈTRE À ÉMISSION OPTIQUE À AFFICHAGE DIRECT
PARTIE I: LE MATÉRIEL

par

T.R. Churchill*

RÉSUMÉ

On retrace brièvement les modifications progressives apportées à un spectromètre à émission optique pour y intégrer un système de commande informatique. Ce système permet de réduire la durée des analyses, d'améliorer la reproductibilité et de traiter les données en direct avec un minimum d'intervention humaine. On présentera dans une deuxième partie le programme de commande. La troisième partie constituera la notice d'utilisation. Elle donnera les instructions détaillées permettant d'analyser des échantillons et de produire des procès-verbaux d'analyse.

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READOUT AND CONTROL SYSTEM FOR A DIRECT READING OPTICAL EMISSION SPECTROMETER

PART I: THE HARDWARE

INTRODUCTION

The direct reading emission spectrometer model 29,000/29,710 universal vacuum air quantometer (UVAQ) used for the elemental analysis of metals and metal alloys was purchased by the Process Metallurgy Research Laboratories (PMRL) of CANMET in 1969 and has been operated by personnel of the Mineral Science Laboratories (MSL) since its intallation. The instrument is capable of measuring 28 elements simultaneously and thus a fast analysis turnaround time which is essential for melt adjustment in foundry operations, can be provided. The readout portion of the spectrometer was the slowest part of the system and it was obvious that the turnaround could be further reduced if this portion of the system was improved. In 1972 it was decided that the system could be best enhanced by the addition of a minicomputer and a more up-to-date electronic measuring system.

The decision to add a minicomputer to the system was based on the fact that in addition to providing faster readout it could also be used to control the various functions of the spectrometer and would allow the immediate calculation of correction factors based on the data being received. In addition to achieving a faster analysis time more reliable results could also be obtained.

The basic minicomputer system was received in November 1974 and this system has been continually modified since that time as funds became available and as new and more efficient devices (peripherals) for the computer appeared on the market. One of the conditions imposed during modifications was that the spectrometer should always remain operational so that there would not be any significant disruption of the foundry operations. This condition resulted in stepwise modifications which slowed the overall conversion process but this has turned out to be an advantage since suitable equipment which was not available in 1974 became available

in later years and this has resulted in a superior overall control and readout system.

Concurrent with the addition of new components to the system it was necessary to write computer programs and to modify these programs when a new component was added. This report will describe the evolution of the hardware portion of the system only and Part II will discuss the computer programs in detail. Part III of the report is an operators manual which will provide the detailed instructions for the operation of the system to provide a sample analysis and analytical report.

OVERVIEW OF SYSTEM EVOLUTION

Figure 1 gives a schematic of the spectrometer system in its original configuration. It can be divided into three main components: 1) source or excitation unit, 2) optical dispersion system, 3) readout and control system. This latter system has undergone the major portion of the modifications and it is shown schematically in more detail in Figure 2 in its original form. The analog signal (current) from each photomultiplier tube (one for each element to be measured) is accumulated in an associated capacitor during the analysis or burn period. The resulting voltage on each capacitor is proportional to the concentration of its associated element and the precise and accurate measurement of this voltage is the function of the readout system. The original system employed a high impedance electrometer in a wheatstone bridge configuration which permitted the measurement of the voltage on the capacitor without discharging it. The main failing of the system was the use of a "dynamic capacitor" to convert the dc voltage on each measuring capacitor to an ac voltage and a motor driven potentiometer to balance the wheatstone bridge. This method as slow and susceptible to vibration and required frequent changing of the motor driven potentiometer because of wear. Because the voltage measurement was slow the scanning mechanism to switch from one capacitor to the next had to be slow and was accomplished by a small bar mounted on a 10 rpm motor and a microswitch which was momentarily closed as each end of the bar brushed past. Thus the maximum measurement rate was one every three seconds. An attempt was made to increase the scan speed to one every 1.5 seconds and then to one every 2 seconds, but this proved to be too fast for the measurement system to handle. The voltage from the motorized potentio-

meter required to balance the wheatstone bridge is equal to the voltage on the capacitor and this voltage was measured with a digital voltmeter (DVM) which was in turn connected to an IBM electric typewriter which provided a printout of the voltage reading. The element concentrations were then read from a table relating voltage readings to concentration. Any necessary corrections were then calculated by the operator.

THE FIRST MAJOR MODIFICATION (ADDITION OF COMPUTER)

The initial computer equipment purchased consisted of a Data General Nova 2/10 minicomputer with 16K words (16 bit) of memory, an ASR 33 teletype and a 12 bit analog to digital convertor (A/D) with a 32 channel multiplexor. In order to connect the equipment to the spectrometer it was necessary to modify the readout portion of the spectrometer and it was decided that this could be accomplished by the Technical Support Group in MSL. Since the spectrometer already had an analog to digital convertor, the DVM, we decided that it could be quickly connected to the computer if we obtained a digital input printed circuit board from Data General which could be plugged directly into the minicomputer. This would allow us to transfer the voltage readings directly to the computer, but still employing the old and slow measuring and scan electronics. However, with this modification it would be possible with a suitable computer program to convert the voltage readings directly to concentrations, to apply corrections and to do some simple statistics on the data. A digital Input/Output (I/O) printed circuit board was purchased and received in January 1975. The voltage levels produced by the DVM were not compatible with the digital I/O board and hence the Technical Support Section built a level convertor which permitted the connection of these two units. At about the same time a three-drive cassette tape magnetic tape unit was added to the system. This addition considerably speeded up the program development since it was no longer necessary to use the slow paper tape reader and punch associated with the teletype. All programs provided by Data General and the programs developed for the system could be stored on the magnetic tape as well as the data collected from the spectrometer. The system as it appeared at this stage of development is diagrammed in Figure 3. Computer programs were written which converted the input data to a suitable format for computational purposes and which printed the reading

for each element selected on the teletype. Also the operator could determine the averages (for each element) of a series of burns, the relative standard deviation and the concentrations by typing suitable three-letter commands on the teletype keyboard. Thus, the first modification eliminated the need for the operator to search through tables to find concentration values and to determine averages by manual calculation.

SECOND MAJOR MODIFICATION (ADDITION OF INTEGRATORS)

The Technical Support Group of MSL designed and built the electronic interface required to replace the existing capacitors and to permit connection to the A/D convertor and the associated multiplexor which was purchased with the computer. The interface consisted of a chassis which contained the necessary power supply for the circuitry and slots with receptacles which permitted the addition of printed circuit boards. The circuits for accumulating the current from each photomultiplier consisted of an operational amplifier in an integrating configuration and eight of these integrators were grouped on one printed circuit board. Also on each board were relays which permitted switching off the input from the photomultipliers and resetting the integrators when the readings were complete (see Figure 4). Four circuit boards each with eight integrators were fabricated and three were installed in the chassis thus providing 24 measurement channels. The fourth board was retained as a spare.

At the completion of a sparking sequence (burn) the voltage on each integrator could be measured either sequentially or randomly by program control. This modification thus eliminated the old measuring and mechanical scanning system and replaced it with a computer controlled system. It also eliminated the scaling potentiometers (two for each channel) which had to be manually adjusted several times each day to maintain the accuracy of measurement (this is referred to as standardization). The new system is capable of measuring the voltage on the integrators at a rate of 28 000 readings per second. The scaling factors can now be calculated directly from measurements made on a secondary standard, stored in the computer memory and applied to each reading prior to storing the result in memory. This change greatly reduced

the standardization time and eliminated the possibility of accidental change of the potentiometers.

As the computer programs to operate the system increased in size and the rate of data collection increased, the limiting factor became the size of available memory. In late 1975 an additional 16K words (32K byte) of memory were added to the computer. While this did not have any effect on the sample analysis time it permitted the addition of some of the useful software routines supplied by the manufacturer such as matrix handling routines for data manipulation and formatting routines for producing more readable hard copy output.

With the above changes to the system it was necessary to produce new calibrations for each element and for each type of matrix. Computer programs were written to assist the calibration procedure; these included a program for collecting both spectrometer readings and certified concentration values, and a linear regression program to determine coefficients for best fit calibration curves from the collected data. Calibrations were done for low alloy steel, cast iron, high alloy steels and tool steel. The computer program used to operate the system in this configuration was being constantly modified and improved as dictated by experience in operating the system.

THIRD MAJOR MODIFICATION (ADDITION OF SOURCE UNIT)

The measurements made with the modified spectrometer were not as reproducible as expected. It was reasoned that the probable cause was the source units used to provide the excitation or vaporization of the samples. When money became available a new source unit of recent design was purchased in 1977. The unit was a Electronically Controlled Waveform Source (ECWS) which uses a thyraton to control the spark repetition rate. The repetition rate is adjustable from 240 breaks/sec to 600 breaks/sec and the RF current is adjustable in the range 4 to 6 amperes. In addition to being adjustable the ECWS operates at 15 000 volts as compared with the 5000 volts obtainable with the old high voltage source unit. Thus we should expect better sampling and more reproducible results. Of course the addition of a new source unit meant that we had to prepare another set of calibration curves. Initially the new source unit was connected into the system in a manner which permitted us to easily switch from the old source

units to the new. Thus we could still provide sample analysis with the old source units and switch to the new unit for calibration. In addition to the expected superiority of the new source unit there is also the possibility that it could be controlled by the computer which could provide improved stability of the source and thus improved reproducibility of the results.

FOURTH MAJOR MODIFICATION (I/O SUBSYSTEM)

The modifications previously described served the purpose of providing a more reproducible data output at a rate much faster than originally possible. The only control of the spectrometer provided by the new components was the stopping of the source unit (when the internal standard channel reached a pre-determined value) and re-setting of the integrators in preparation for the next analysis. The original control system was still in use and provided the following functions:

1) Control of argon gas flow
2) Starting of voltage integration after a pre-set delay
3) Start of voltage integration after a pre-set delay
4) Switching off and on of grating lamp
5) Timing of above functions

In addition to the above functions there is an important procedure referred to as "profiling" which is used to maintain the optical system in optimum alignment. The operator has to connect two or more channels to the original readout system to carry out this procedure. All the controls described above are accomplished with an outdated electromechanical system which consists of three electrical timer switches and a multitude of multi-contact relays. The relays require frequent adjustment and cleaning to keep them in satisfactory working condition. It was our aim from the outset to use the computer system to provide the timing and control functions required by the spectrometer and to replace the relays with solid state devices.

In 1981 Data General made available a system (called an I/O subsystem) which was ideally suited to our requirements. The system is plug compatible with our computer and allows the selection of a wide variety of plug-in circuit cards which permits us to assemble a system to satisfy our requirements. The basic components of the I/O subsystem and a set of three modules were ordered and received in late 1981. The initial printed circuit cards we obtained were:

1) General Purpose Input Module (16 inputs)
2) Triac Output Module (16 outputs)
3) Digital to Analog Converter (4 outputs)

The chassis and power supply will accommodate a total of sixteen printed circuit boards which provides plenty of scope for future additions to the system. Figure 5 is a schematic of the complete system in its current configuration.

The push of the start button on the spectrometer now signals the computer that an analysis is to be started and from that point in time the computer will control the sequencing of the various functions as well as the collection of the data. The operator controls the manipulation of all the data via function keys on the console input keyboard. There are currently twelve functions available to the operator and these are described in detail in Part II of this series of reports on the emission spectrometer system.

DISCUSSION

The spectrometer readout and control system in its present configuration provide all the improvements which were aimed for in the design phase:

1) Decreased analysis time
2) Better reproducibility
3) Improved reliability and ease of operation

The analysis time has been reduced from six (6) minutes per sample to two (2) minutes per sample (these figures are based on triplicate determinations). The analysis time is now practically independent of the number of elements being determined (typically this number is 10) because increasing the number of elements merely adds a few seconds of printout time. The old system required the operator to search through a separate table relating the reading to element concentration for each additional element. Also corrections to the data are immediately applied by the analysis program and do not require separate calculations by the operator as was necessary with the original system.

In addition to time reduction for the routine analyses the time required for procedures such as standardization, calibration, homogeneity testing and statistical analysis of the data has been greatly reduced because the data can be manipulated immediately or stored for later use without the necessity of re-entering it. The analysis time could be further decreased by reducing the burn or sparking time from the current 20 seconds to 15 or even 10 seconds but this would result in decreased reproducibility.

The reproducibility expressed as per cent relative standard deviation (% RSD) has been reduced from the range two (2) to five (5) per cent to the range 0.3 to one (1) per cent. The range of values arises because the more volatile elements such as carbon, sulphur, phosphorous and boron give poorer reproducibility with the spark excitation technique. The accuracy of measurement is not significantly changed because it is highly dependent on the availability of suitable standards to calibrate the instrument. The improved reproducibility does permit the detection of sample inhomogeneity with a greater degree of certainty.

The reliability of the instrument has been greatly improved as a result of replacing the relays with solid state switches and elimination of electro-mechanical voltage measuring arrangement. The amount of down-time with the new system has amounted to a few hours in the past two years which has permitted a higher sample throughput. There is now less chance of operator error because it is no longer necessary to adjust the myriad of potentiometers (64 in total) or to have one accidentally changed as had happened frequently. The operator does not have to search through numerous tables to find concentration values or to apply corrections by separate calculations.

It would be possible to improve the system by providing monitoring and control of the source or excitation unit and by changing the method of signal detection from an integrating to an instantaneous method. The first change should improve the reproducibility and the second would provide the capability of measuring the free and bound portions of some of the elements (such measurements have metallurgical significance). Both of these changes would require the design and construction of relatively complex electronic circuits and a major modification of our present system and we are not currently planning to undertake them.

ACKNOWLEDGEMENTS

A great deal of credit for the successful completion of this work must go to G. Alexander and P. Richer of the Technical Support Group. They undertook the design and construction of the various computer to spectrometer interfaces required and made valuable suggestions during the course of the modifications.

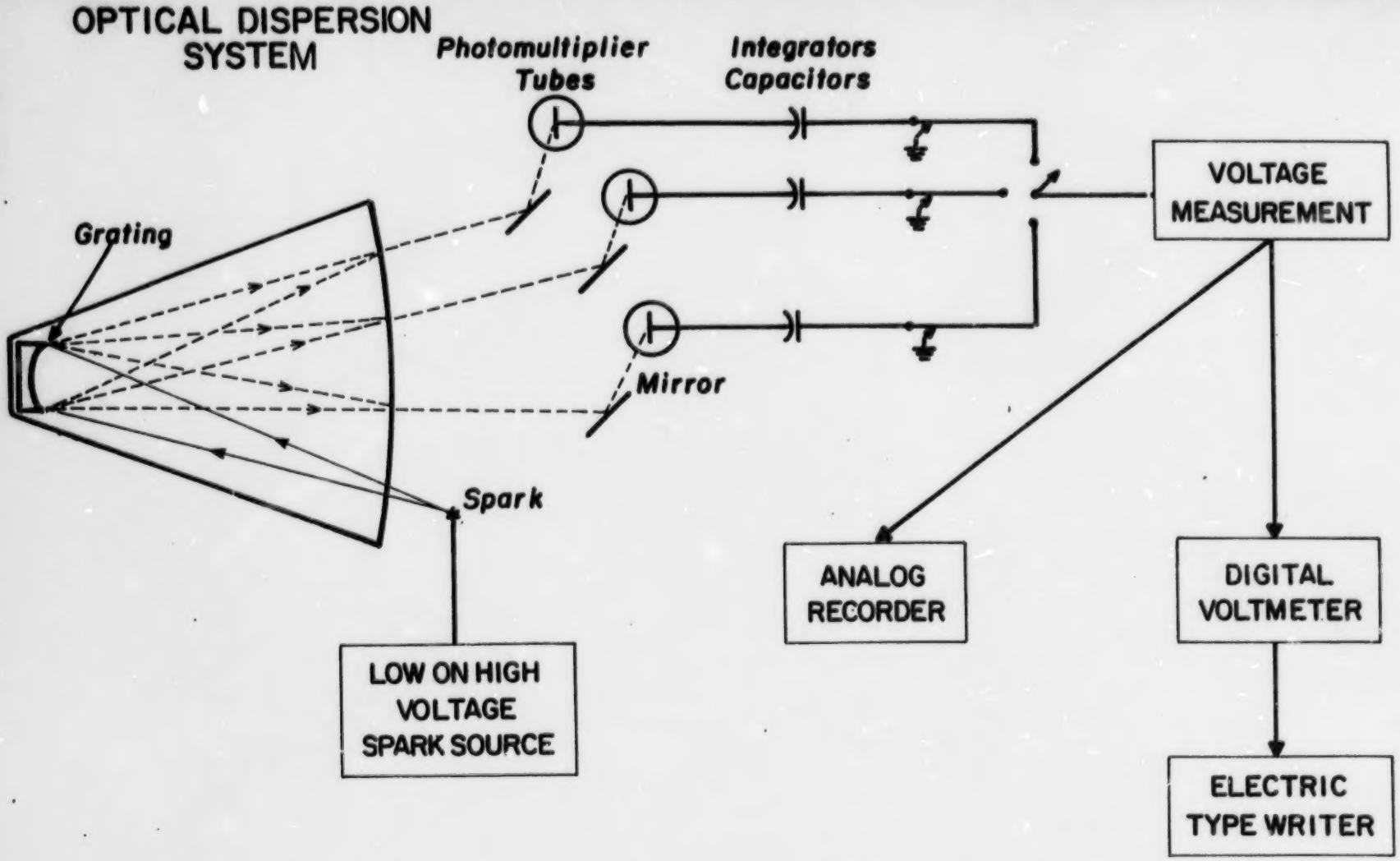

FIG. 1. SCHEMATIC OF SPECTROMETER SYSTEM

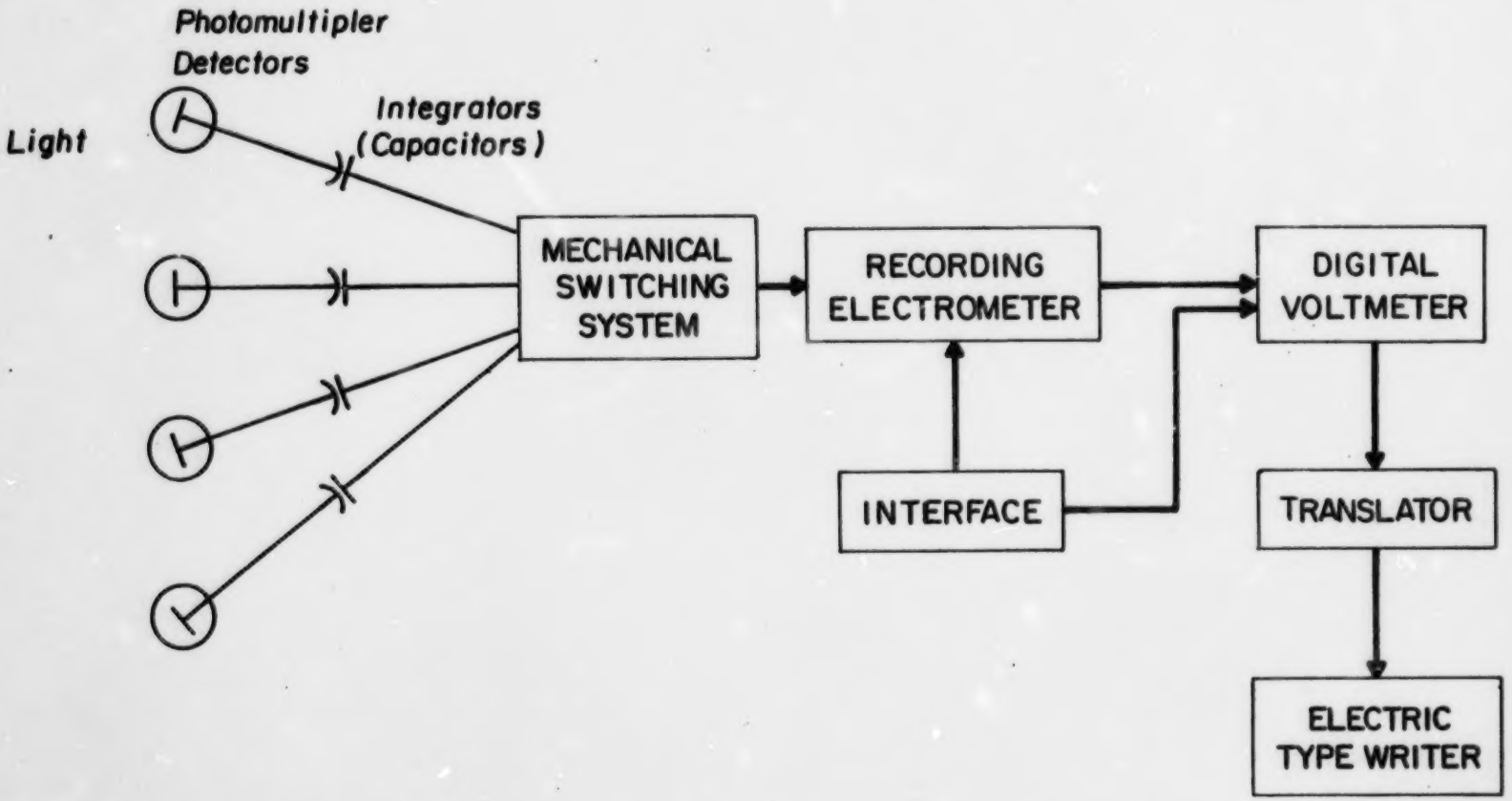

FIG. 2. READOUT SYSTEM BEFORE MODIFICATION

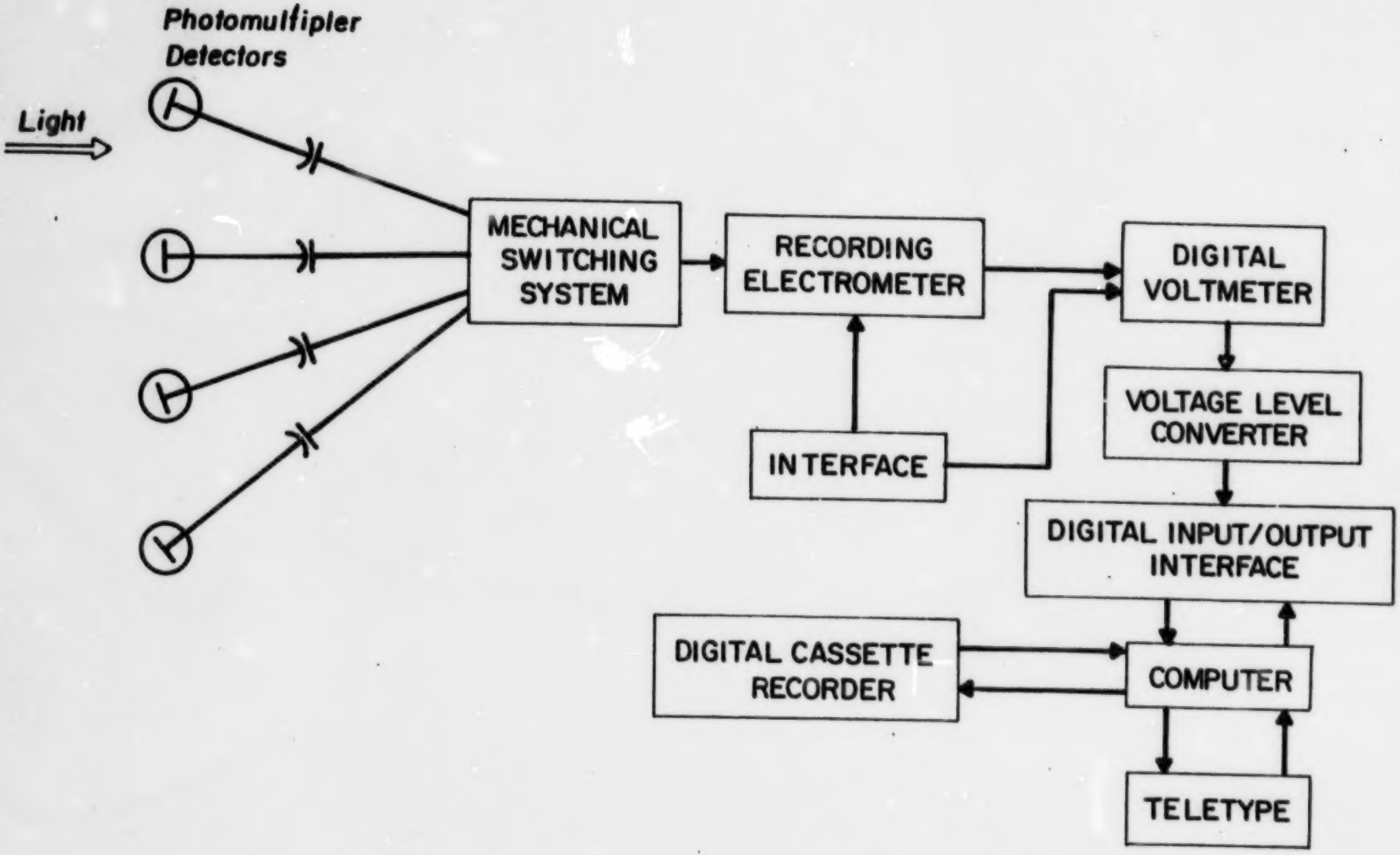

FIG. 3. READOUT SYSTEM WITH COMPUTER

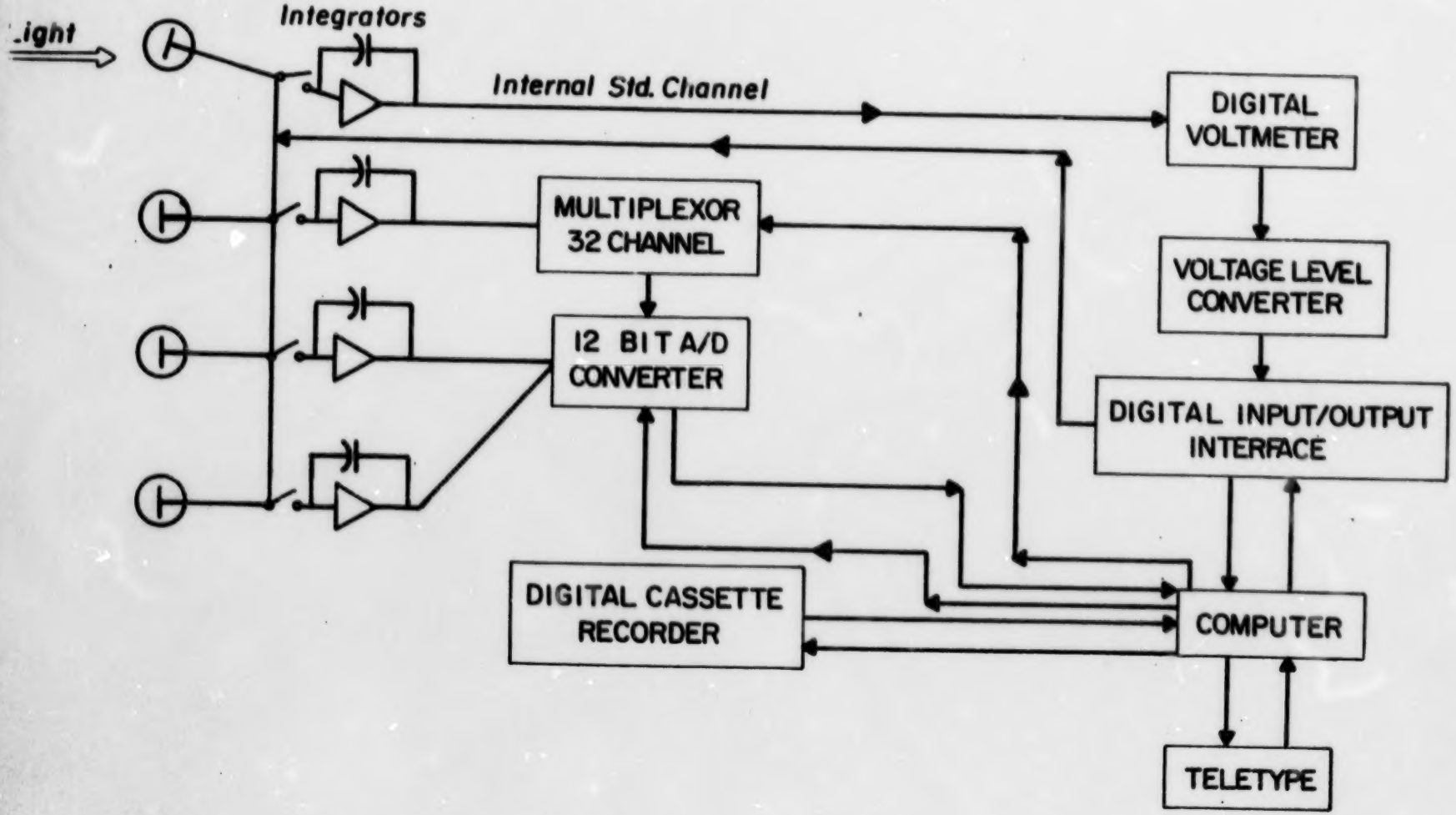

FIG. 4. COMPUTER READOUT WITH ELECTRONIC SWITCHING

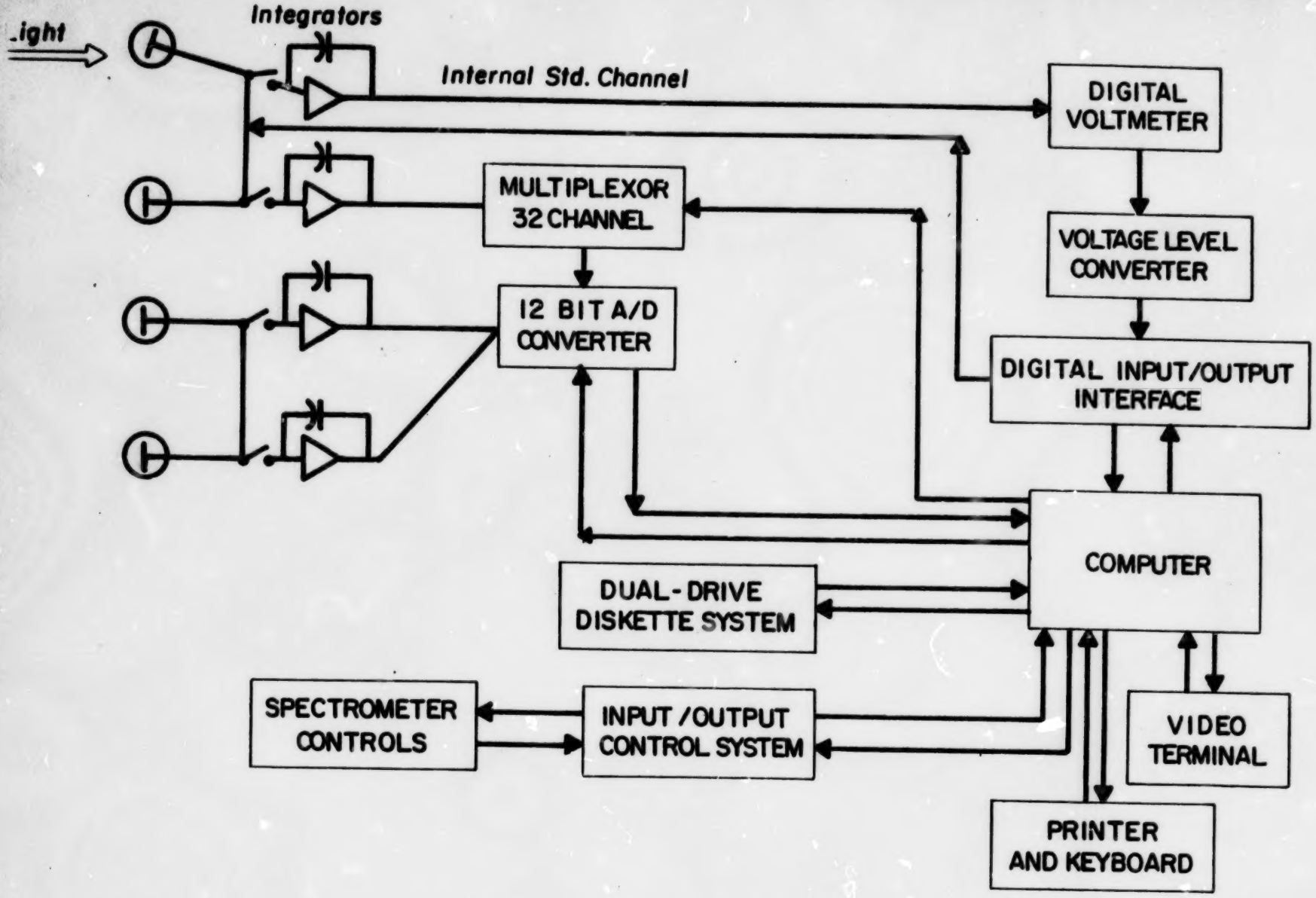

FIG. 5. FINAL READOUT AND CONTROL SYSTEM